1983

# Report on Recommended Specifications for Microchemical Apparatus

## Van Slyke Manometric Apparatus

*Committee on Microchemical Apparatus, Division of Analytical Chemistry, American Chemical Society*

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In accordance with the practice followed in previous reports of the Committee on Microchemical Apparatus (*2–4*), these specifications are for pieces of apparatus that are either the most widely used in their respective fields of application or are an improvement over such apparatus according to tests made by members of this committee and co-operating chemists.

In this report, specifications are recommended for the conventional Van Slyke manometric apparatus (*9, 11, 14, 20*) used in connection with the following determinations: carbon (*11, 14*), primary amino nitrogen in the aliphatic α-amino acids (*11–13, 20*), indirect methods which are based on the combustion of organic precipitates (*6–8, 18, 21*), and blood and urine analyses (*1, 5, 9, 10, 13, 15–17, 19, 20*). Types of apparatus employing spherical joints on the extraction chamber and employing mag-

netic stirring are not included, due to the fact that patents are involved. The micromanometric apparatus (*22*) recently described is also not included, because these pieces have not yet received widespread acceptance.

Figure 1 shows the extraction chamber graduated at the 0.5-, 2.0-, and 50-ml marks. This piece is used exclusively for the determination of nitrogen (*9*, *11–13*, *19*, *20*), while the extraction chamber shown in Fig. 2, which is graduated at the 0.5-, 2.0-, 10.0-, and 50-ml marks, may be used for either the determination of carbon or nitrogen (*11*, *14*). Both extraction chambers require water jackets (*9*, *11*, *14*, *20*). No recommended specifications are given for these jackets, inasmuch as they would have to be individually fitted to the apparatus involved. The tolerances specified for the extraction chambers are based on starting each calibration at the intersection of the stopcock plug and the capillary tube.

Figure 3 shows the manometer. This is connected by means of the spherical joint to the bent tube with stopcock which, in turn, is connected to the extraction chamber. Recommended specifications for this connecting tube are not given, inasmuch as its dimensions depend upon the type of mounting on individual support stands. In addition, a standard-type leveling bulb (250-ml capacity) and flexible tubing for connecting this bulb to the above-mentioned connecting tube with stopcocks are required.

Figures 4 and 5 show pipettes (*9*, *11*) with and without stopcocks, respectively, which are used to make additions to the extraction chamber. Figure 6 shows a blow-out type pipette (*9*).

Figure 7 shows the Hempel pipette (*9*, *11*, *13*) used in the determination of nitrogen, while Fig. 8 shows the alkali storage tube (*11*, *14*) used for the addition of alkaline hydrazine to the extraction chamber in the determination of carbon.

Figure 9 shows the combustion assembly (*11*, *14*) used in the determination of carbon. It consists of the connecting tube (with cup and stopcock) and the combustion tube. For the indirect methods based on the combustion of organic precipitates, the centrifuge combustion tubes shown in Figs. 10 and 11 are used in place of the combustion tube shown in the assembly. The centrifuge combustion tube shown in Fig. 10 is used in the determination of magnesium (*7*), phosphorus (*6*, *8*), and sulfur (*6*), while that shown in Fig. 11 is used in the determination of calcium (*18*).

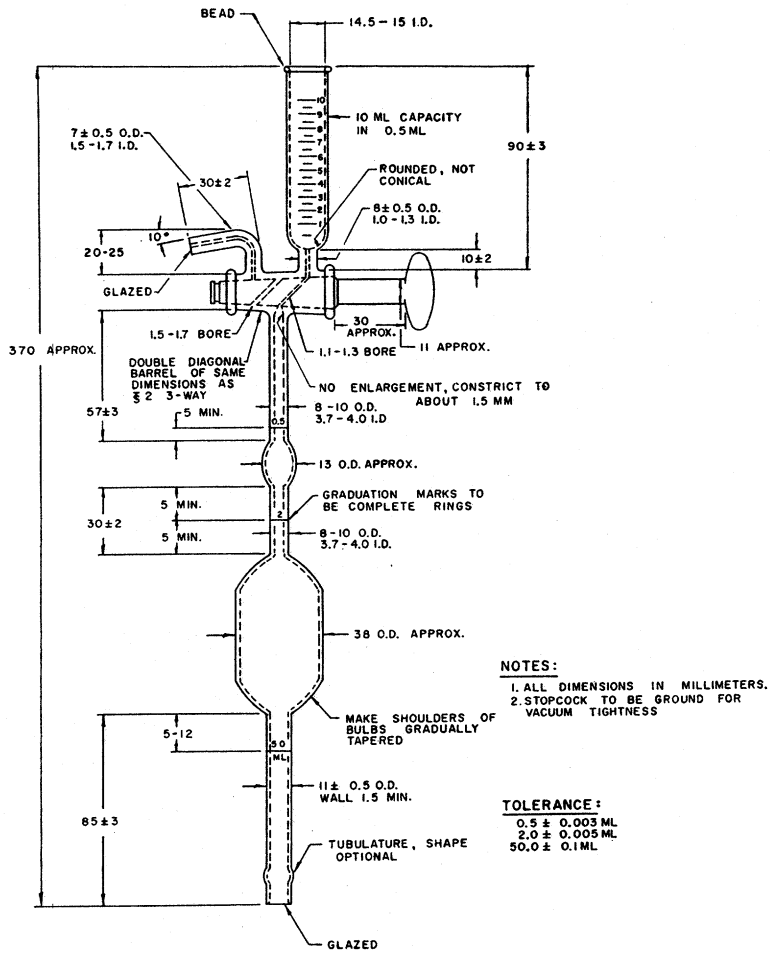



FIG. 1. Van Slyke extraction chamber.

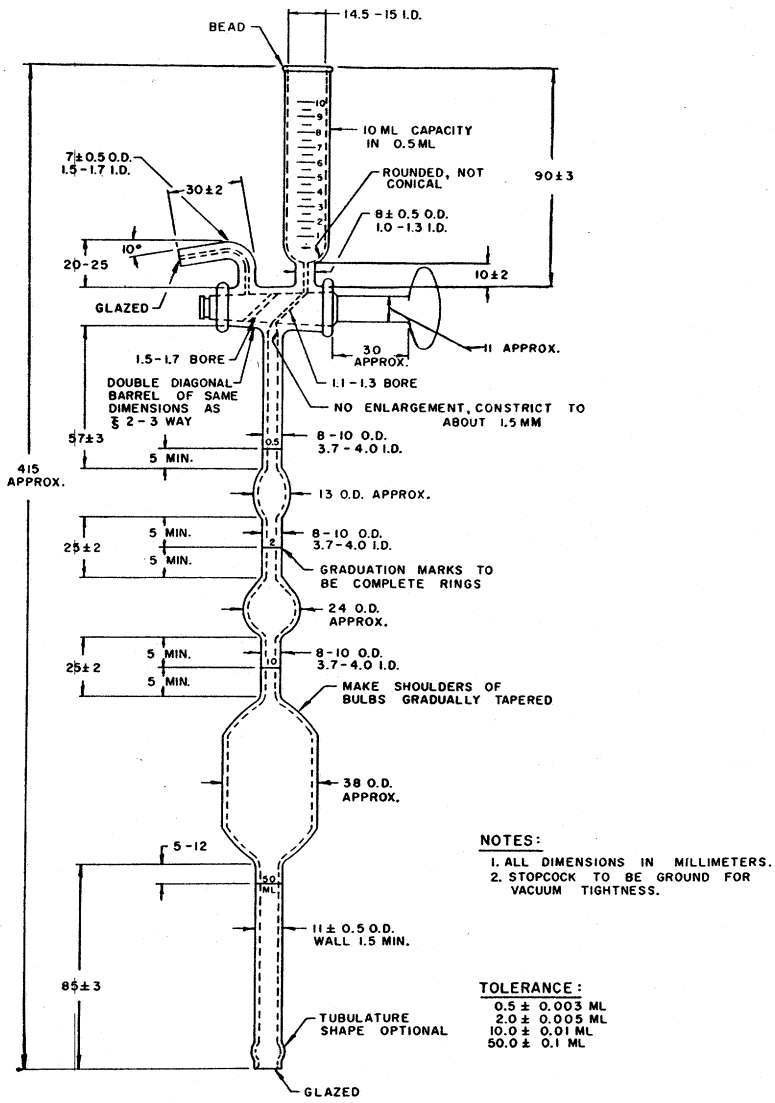



FIG. 2. Van Slyke extraction chamber.

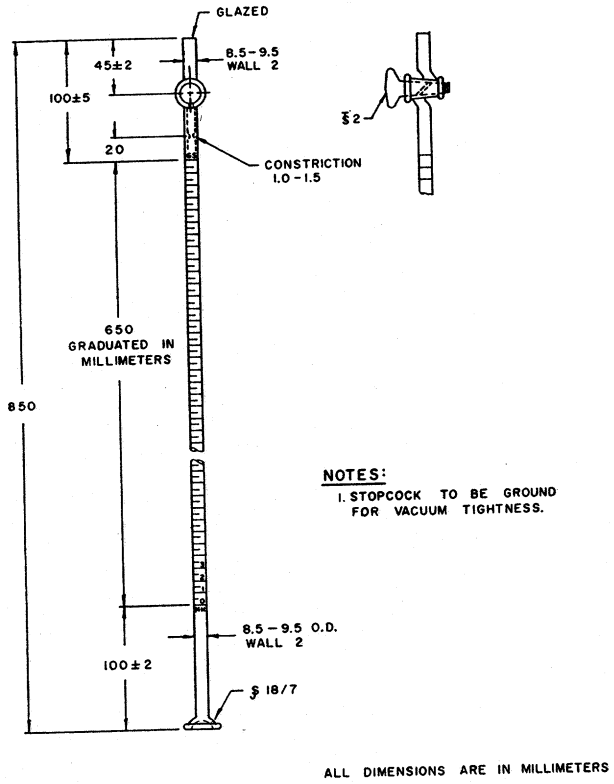
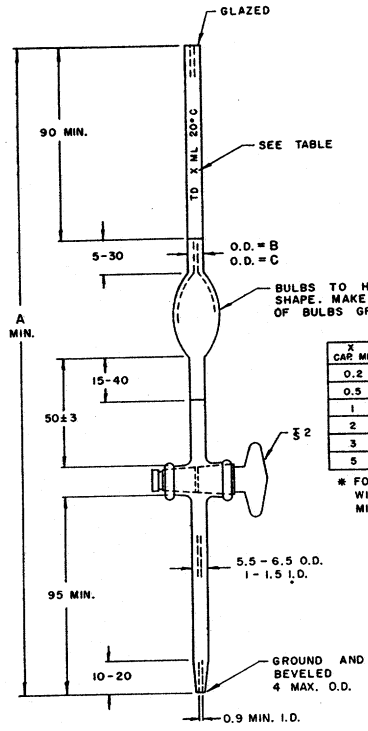
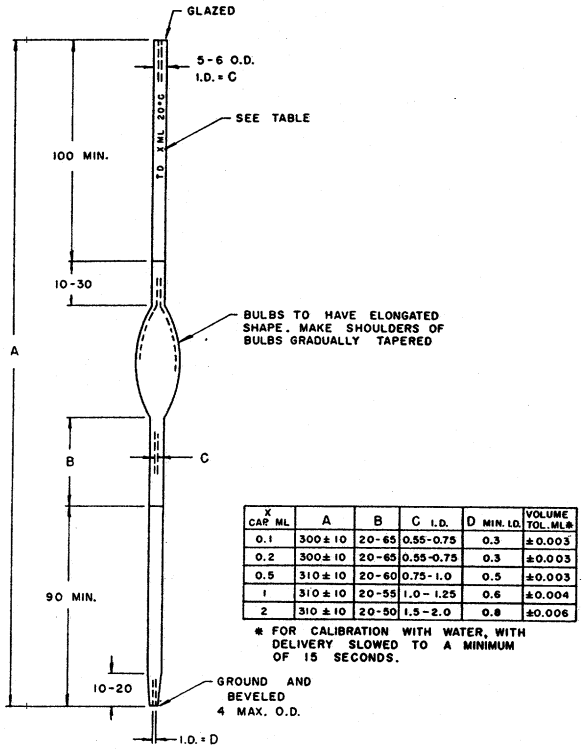



FIG. 3. Manometer.




| X CAP ML | A | B | C | VOLUME TOL. ML * |
|---|---|---|---|---|
| 0.2 | 290 | 5-6 | 0.55-0.75 | ±0.003 |
| 0.5 | 290 | 5-6 | 0.75-1.0 | ±0.003 |
| 1 | 300 | 6-7 | 1.75-2.0 | ±0.004 |
| 2 | 310 | 6-7 | 1.75-2.0 | ±0.006 |
| 3 | 310 | 6-7 | 1.75-2.0 | ±0.010 |
| 5 | 310 | 6-7 | 1.75-2.0 | ±0.010 |

* FOR CALIBRATION WITH WATER, WITH DELIVERY SLOWED TO A MINIMUM OF 15 SECONDS.

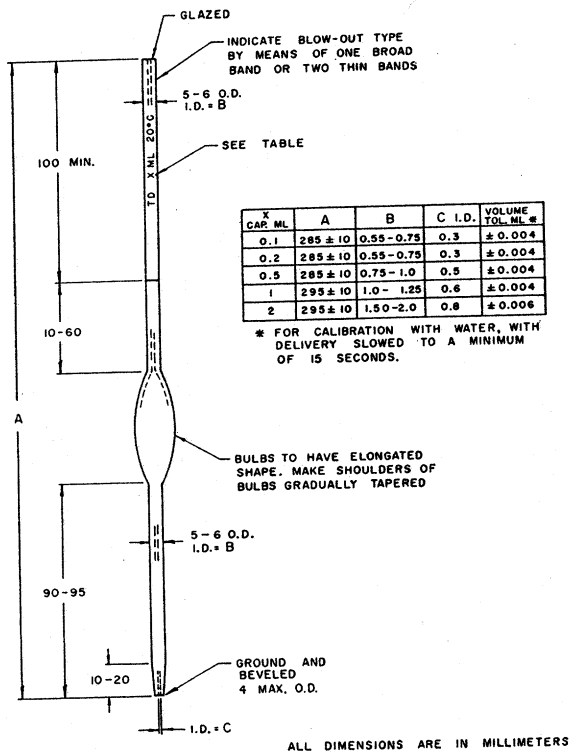



| X CAP. ML | A | B | C I.D. | D MIN. I.D. | VOLUME TOL. ML* |
|---|---|---|---|---|---|
| 0.1 | 300 ± 10 | 20-65 | 0.55-0.75 | 0.3 | ±0.003 |
| 0.2 | 300 ± 10 | 20-65 | 0.55-0.75 | 0.3 | ±0.003 |
| 0.5 | 310 ± 10 | 20-60 | 0.75-1.0 | 0.5 | ±0.003 |
| 1 | 310 ± 10 | 20-55 | 1.0-1.25 | 0.6 | ±0.004 |
| 2 | 310 ± 10 | 20-50 | 1.5-2.0 | 0.8 | ±0.006 |

* FOR CALIBRATION WITH WATER, WITH DELIVERY SLOWED TO A MINIMUM OF 15 SECONDS.

ALL DIMENSIONS ARE IN MILLIMETERS

FIG. 5. Van Slyke pipettes without stopcock.




| X CAP. ML | A | B | C I.D. | VOLUME TOL. ML * |
|---|---|---|---|---|
| 0.1 | 285 ± 10 | 0.55-0.75 | 0.3 | ± 0.004 |
| 0.2 | 285 ± 10 | 0.55-0.75 | 0.3 | ± 0.004 |
| 0.5 | 285 ± 10 | 0.75-1.0 | 0.5 | ± 0.004 |
| 1 | 295 ± 10 | 1.0-1.25 | 0.6 | ± 0.004 |
| 2 | 295 ± 10 | 1.50-2.0 | 0.8 | ± 0.006 |

* FOR CALIBRATION WITH WATER, WITH DELIVERY SLOWED TO A MINIMUM OF 15 SECONDS.

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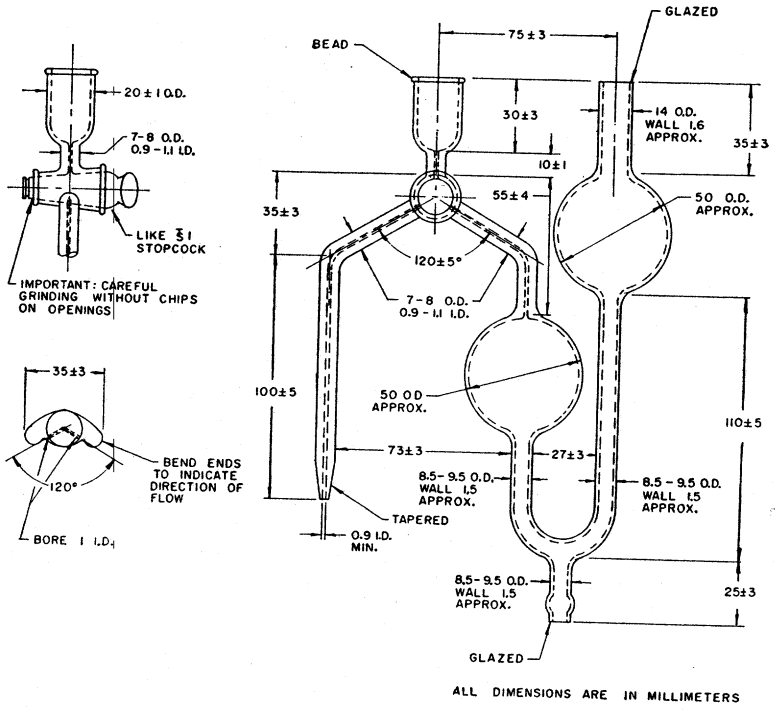


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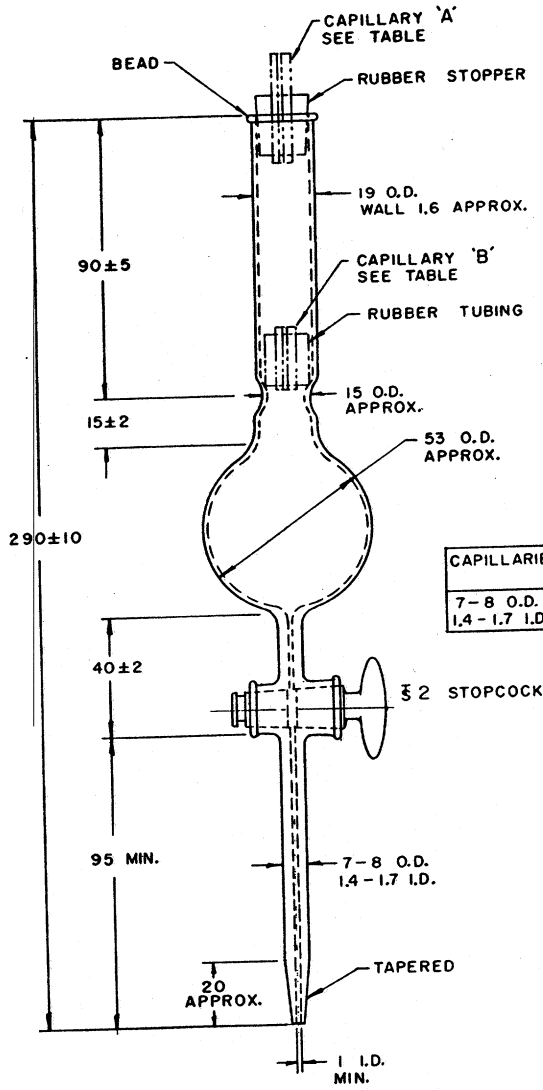


| CAPILLARIES | LENGTH A | LENGTH B |
|---|---|---|
| 7-8 O.D. 1.4-1.7 I.D. | 35±3 | 20±2 |

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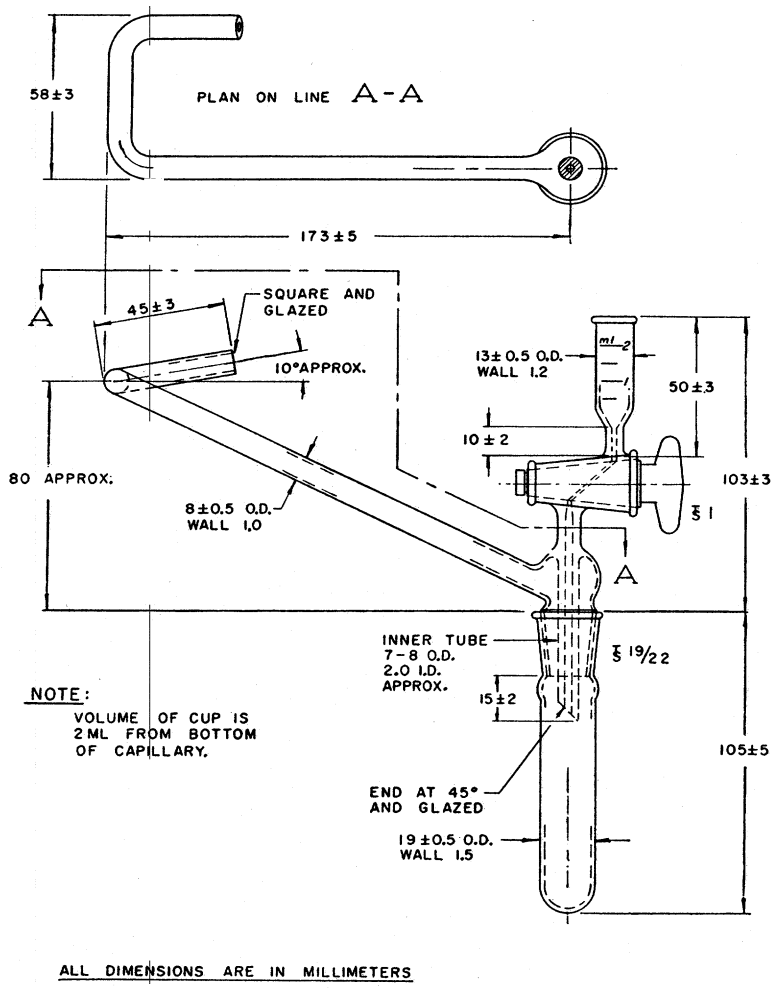


FIG. 9. Van Slyke combustion assembly.

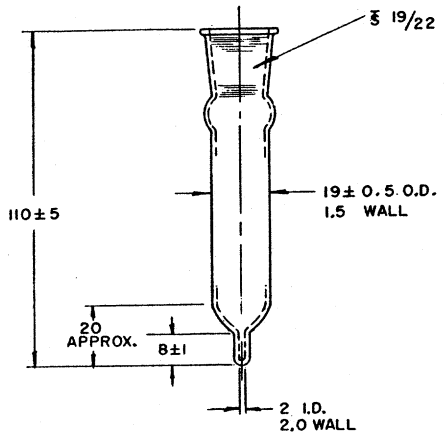


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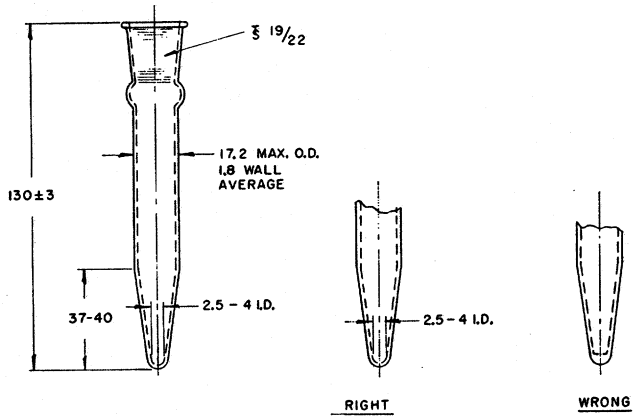
FIG. 10. Van Slyke centrifuge combustion tube.




ALL DIMENSIONS ARE IN MILLIMETERS

## REFERENCES


1. AUSTIN, J. H., A note on the estimation of carbon dioxide in serum in the presence of ether by the Van Slyke method. *J. Biol. Chem.* **61**, 345-353 (1924).
2. COMMITTEE ON MICROCHEMICAL APPARATUS, DIVISION OF ANALYTICAL CHEMISTRY, AMERICAN CHEMICAL SOCIETY, 1953 Report on recommended specifications for microchemical apparatus. Weighing and drying. *Anal. Chem.* **26**, 1186-1190 (1954); Report on recommended specifications for microchemical apparatus. Alkoxyl. *Anal. Chem.* **28**, 112-115 (1956); Report on recommended specifications for microchemical apparatus. Volumetric glassware. Flasks, pipets, and centrifuge tubes. *Anal. Chem.* **28**, 1993-1995 (1956); Report on recommended specifications for microchemical apparatus. Volumetric glassware. Microliter pipets. *Anal. Chem.* **30**, 1702-1703 (1958); Report on recommended specifications for microchemical apparatus. Volumetric glassware. Micropipets. *Anal. Chem.* **32**, 1045-1046 (1960); Report on recommended specifications for microchemical apparatus. Oxygen combustion flask. *Anal. Chem.* **33**, 1789-1790 (1961).
3. COMMITTEE FOR THE STANDARDIZATION OF MICROCHEMICAL APPARATUS, DIVISION OF ANALYTICAL CHEMISTRY, AMERICAN CHEMICAL SOCIETY, All-metal needle valve of the Hershberg-Southworth type. *Anal. Chem.* **21**, 1283-1284 (1949); 1949 Report on recommended specifications for microchemical apparatus. Carbon-hydrogen, Dumas nitrogen, sulfur, and halogen. *Anal. Chem.* **21**, 1555-1565 (1949); New design of rubber stoppers for microchemistry. *Anal. Chem.* **22**, 1228 (1950); Recommended specifications for microchemical apparatus. Micro-Kjeldahl nitrogen. *Anal. Chem.* **23**, 523-528 (1951); Tabulation of apparatus used for the micro-Dumas determination. *Anal. Chem.* **23**, 537-538 (1951); Recommended specifications for microchemical apparatus. Carius method. *Anal. Chem.* **23**, 1689 (1951).
4. EDITORIAL NOTE, Standardization of microchemical apparatus, *Chem. Eng. News* **26**, 883 (1948).
5. HARINGTON, C. R., AND VAN SLYKE, D. D., On the determination of gases in blood and other solutions by vacuum extraction and manometric measurement. II. *J. Biol. Chem.* **61**, 575-584 (1924).
6. HOAGLAND, C. L., Microdetermination of sulfate and phosphate by manometric combustion of their organic precipitates. *J. Biol. Chem.* **136**, 543-551 (1940).
7. HOAGLAND, C. L., Micromanometric determination of magnesium. *J. Biol. Chem.* **136**, 553-558 (1940).
8. KIRK, E., Gasometric microdetermination of phosphoric acid. *J. Biol. Chem.* **106**, 191-201 (1934).
9. PETERS, J. P., AND VAN SLYKE, D. D., "Quantitative Clinical Chemistry," Vol. 2. Williams & Wilkins, Baltimore, Maryland, 1943.
10. SENDROY, J., JR., AND LIU, S. H., Gasometric determination of oxygen and carbon dioxide in blood. *J. Biol. Chem.* **89**, 133-152 (1930).
11. STEYERMARK, AL, "Quantitative Organic Microanalysis," 2nd ed., pp. 454-494. Academic Press, New York, 1961.
12. VAN SLYKE, D. D., Gasometric micro-Kjeldahl determination of nitrogen. *J. Biol. Chem.* **71**, 235-248 (1927).

13. VAN SLYKE, D. D., Manometric determination of primary amino nitrogen and its application to blood analysis. *J. Biol. Chem.* **83**, 425-447 (1929).
14. VAN SLYKE, D. D., AND FOLCH, J., WITH THE ASSISTANCE OF PLAZIN, J., Manometric carbon determination. *J. Biol. Chem.* **136**, 509-541 (1940).
15. VAN SLYKE, D. D., AND HAWKINS, J. A., A gasometric method for determination of reducing sugars, and its application to analysis of blood and urine. *J. Biol. Chem.* **79**, 739-767 (1928).
16. VAN SLYKE, D. D., AND HILLER, A., Gasometric determination of hemoglobin by the carbon monoxide capacity method. *J. Biol. Chem.* **78**, 807-819 (1928).
17. VAN SLYKE, D. D., AND KIRK, E., Comparison of gasometric, colorimetric, and titrimetric determinations of amino nitrogen in blood and urine. *J. Biol. Chem.* **102**, 651-682 (1933).
18. VAN SLYKE, D. D., AND KREYSA, F. J., Microdetermination of calcium by precipitation as picrolonate and estimation of the precipitated carbon by manometric combustion. *J. Biol. Chem.* **142**, 765-776 (1942).
19. VAN SLYKE, D. D., AND KUGEL, V. H., Improvements in manometric micro-Kjeldahl and blood urea methods. *J. Biol. Chem.* **102**, 489-497 (1933).
20. VAN SLYKE, D. D., AND NEILL, J. M., The determination of gases in blood and other solutions by vacuum extraction and manometric measurement. I. *J. Biol. Chem.* **61**, 523-573 (1924).
21. VAN SLYKE, D. D., PAGE, I. H., AND KIRK, E., A manometric micromethod for determination of carbon in organic compounds. *J. Biol. Chem.* **102**, 635-649 (1933).
22. VAN SLYKE, D. D., AND PLAZIN, J., "Micromanometric Analyses," Williams & Wilkins, Baltimore, Maryland, 1961.